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## PATENT ABSTRACTS OF JAPAN

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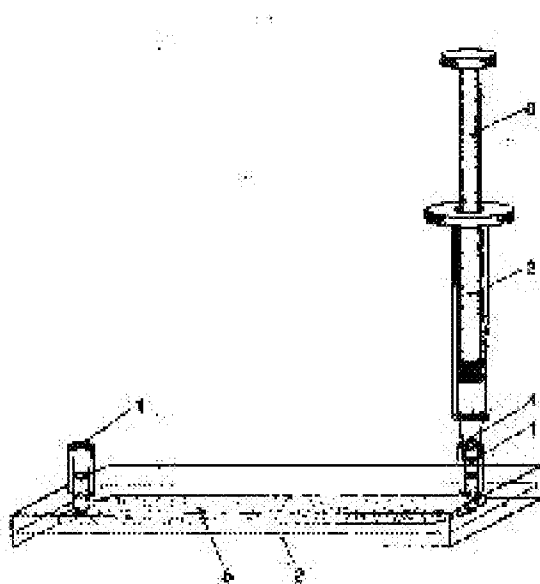
(72)Inventor : **SAWADA TSUTOMU**

**(54) VESSEL FOR PRODUCING COLLOIDAL SINGLE CRYSTAL, METHOD FOR TURNING COLLOIDAL CRYSTAL INTO SINGLE CRYSTAL BY USING THE SAME, AND METHOD FOR PRODUCING DRIED COLLOIDAL CRYSTAL**

(57)Abstract:

**PROBLEM TO BE SOLVED:** To provide a means for producing a colloidal single crystal, by which mixing of air bubbles into a vessel when a colloidal solution is injected can be prevented, the formed colloidal single crystal is prevented from being disordered when a liquid injector is taken out after forming the single crystal and further the formed single crystal can be uniformly dried when the colloidal single crystal is produced by allowing the colloidal solution to flow in a uniaxial direction and imparting shear flow to the colloidal solution in a vessel having a flat plate-shaped capillary.

**SOLUTION:** In the vessel 2 for producing the colloidal single crystal, which has liquid injection ports 1 provided at the both ends of the vessel in a protruding condition and the flat plate-shaped capillary 5 having a gap of  $\leq 0.5$  mm, the structure of each liquid injection port 1 is set to have a taper in such a manner that the opening part of the entrance side is wider and



the opening part of the back side is narrower. The vessel is made of a transparent plastic having a certain moisture permeability, and a colloidal solution using water as a dispersion medium is used as the colloidal solution.

JAPANESE

[JP,2003-212700,A]

CLAIMS DETAILED DESCRIPTION TECHNICAL  
FIELD PRIOR ART EFFECT OF THE INVENTION  
TECHNICAL PROBLEM MEANS EXAMPLE  
DESCRIPTION OF DRAWINGS DRAWINGS

[Translation done.]

\* NOTICES \*

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**CLAIMS**

[Claim(s)]

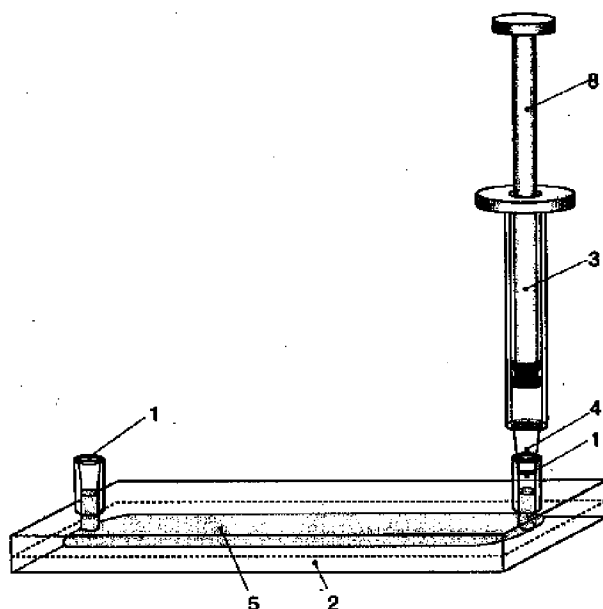
[Claim 1]It is a colloid single crystal production container which has a plate shaped capillary which has the pouring-in mouth provided in container both ends by protruding, and has a gap of 0.5 mm or less succeeding this pouring-in mouth, A colloid single crystal production container, wherein it attaches and sets up a taper narrowly and a capillary tube field at least is widely constituted [ structure / of this pouring-in mouth / opening / entrance-side ] with a transparent material in the back as for construction material of a container.

[Claim 2]The colloid single crystal production container according to claim 1, wherein the container according to claim 1 is glass.

[Claim 3]A colloid single crystal production container, wherein the container according to claim 1 is a product made from a plastic.

[Claim 4]A manufacturing method of a colloid single crystal single-crystal-izing a colloid crystal by pressing a colloidal solution fit in the colloid single crystal production containers according to claim 1, 2, or 3, and giving a

Drawing selection **Representative draw**



[Translation done.]

shearing flow to this colloidal solution.

[Claim 5]A manufacturing method of a dry colloid crystal pressing a colloidal solution fit in the colloid single crystal production containers according to claim 1 or 2, single-crystal-izing a colloid crystal by giving a shearing flow to this colloidal solution, and giving subsequently to a drying process.

[Claim 6]Press a colloidal solution fit in the colloid single crystal production containers according to claim 3, and a colloid crystal is single-crystal-ized by giving a shearing flow to this colloidal solution, Subsequently, a manufacturing method of a dry colloid crystal emitting moisture slowly by slight moisture permeability which the plastic material has, and obtaining a dry crystal by this.

[Claim 7]After forming in a colloid single crystal a colloidal solution which makes water carrier fluid by giving a shear flow in a plate shaped capillary container of plastic material, by slow discharge of moisture using the slight moisture permeability of the plastic material. A manufacturing method of a dry colloid crystal drying a colloid single crystal.

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[Translation done.]

JAPANESE

[JP,2003-212700,A]

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## DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

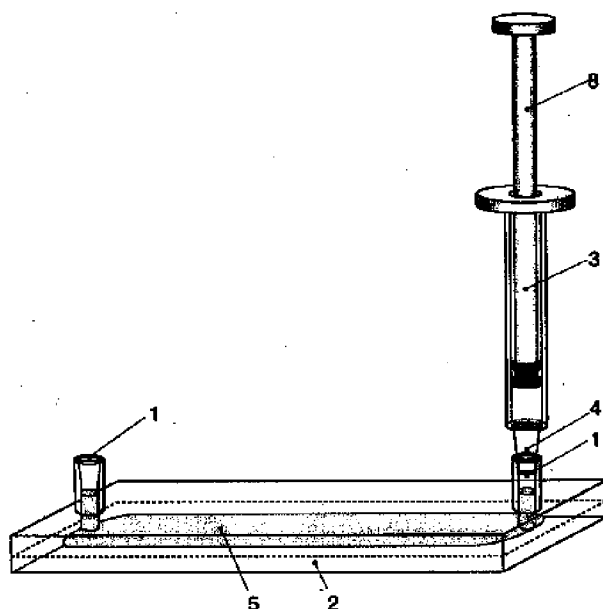
[Field of the Invention]This invention relates to the single crystal-ized method of the colloid crystal which manufactures a colloid single crystal using the colloid single crystal production container and this container for saving the crystal which produced the colloid single crystal which can serve as an optical element material, and was produced, and the manufacturing method of a dry colloid crystal.

[0002]

[Description of the Prior Art]In the colloidal solution which distributed particles, such as polymers RATTEKUSU and silica, to liquid media, such as water and an organic solvent, Under a relevant condition, the structure which particles arranged regularly in three dimensions is formed spontaneously, and the colloidal solution in such a state is called the colloid crystal (reference: "colloid science I"119 page edited by the Chemical Society of Japan), and attracts attention from the use as an optical material.

[0003]Usually, although a colloid crystal is obtained by the polycrystalline state in which crystal domains with a size of

Drawing selection **Representative draw**



[Translation done.]

several millimeters or less gathered, In this invention person etc., by giving a shearing flow to a colloidal solution, it succeeded in making the huge single crystal domain which attains to cm size form in a plate-like capillary tube, and patent application was previously carried out based on this (application for patent No. 217660 [ 2000 to ]).

[0004]

[Problem(s) to be Solved by the Invention]Old art, i.e., the outline of the above-mentioned prior invention, As shown in drawing 1, the cylindrical pouring-in mouth for pouring in a colloidal solution protrudes on a container external wall, and is provided in container both ends, In and the colloid single crystal production containers with which the capillary tube space which was connected with this cylindrical pouring-in mouth and presented the narrow plate-like gap is provided. The colloidal solution which is in a colloid crystal state from the pouring-in mouth of the end is poured in, Fill the inside of a container with a colloidal solution, and, subsequently to a colloidal solution, a shearing flow is given, Then, after making 1 shaft orientations flow in parallel with a substrates face in the capillary tube space which presented the plate-like gap, it is made to stand it still and the colloid single crystal which is a colloid crystal which has the organization excellent in three-dimensional homogeneity is made to form. From pouring in of a colloidal solution in the meantime, a series of operations of resulting in grant of a shearing flow, and a flow of 1 shaft orientations, First, fill a colloidal solution to a syringe (injector) with a needle, and a needle, Insert in a cylindrical pouring-in mouth and a colloidal solution is poured in from a pouring-in mouth by piston operation of a syringe, The empty syringe which does not attach a needle was sealed and attached to the pouring-in mouth by passing a tube after pouring-in operation completion (drawing 1), and the pressure for a shearing flow was put on the colloidal solution in a container, i.e., the sample solution in a capillary tube.

[0005]However, this method had a problem in that it is easy to mix air bubbles in a capillary tube in the process in which a sample colloidal solution is poured in a container so that it might mention later. Removing was difficult for the air bubbles once mixed in the capillary tube, and they had become an obstacle in the single crystal-ized processing by flow following this operation. (Technical problem 1)

[0006]Although this syringe is sampled from a pouring-in mouth after completing a series of syringe operations of obtaining a crystal, If it tries to remove a syringe the whole tube from a tube after the process operation which the tip of the syringe is connected to the pouring-in mouth via the tube by the old method as described above (drawing 1), and obtains a single crystal, Pressure fluctuation started the

capillary part and there was inconvenience of disturbing the state of the once single-crystal-ized sample. (Technical problem 2)

[0007]In order to obtain the dry colloid crystal of the good big domain of single crystal nature, on the whole, it is necessary to dry this uniformly very slowly after forming a single crystal by giving a shearing flow in a plate shaped capillary. A dry colloid crystal is compared with the colloid crystal in which the solvent exists, a photonic band gap (reference: -- work besides J.D.Joannopoulos.) since the difference of the refractive index of particles and the other portion is large It is expected that it is connected with the application development as a bigger optical element material excellent in functionality than that [ Fujii and Inoue translation "photonic crystal" Corona Publishing ].

[0008]However, although the drying process after forming a single crystal and sampling a syringe subsequently in the old method, i.e., the method of the above-mentioned prior invention, was performed by the mode dried by opening wide the pouring-in mouth provided in both ends, and evaporating a solvent from there, It was very difficult not to obtain a colander in this drying method, if it is in the place near an open end, and the direction of the back more distant than this at unevenness, but to obtain the high dry colloid crystal of single crystal nature based on a uniform drying rate. (Technical problem 3)

[0009]Also in the process of pouring a colloidal solution in a container in order that this invention may obtain a colloid single crystal, Also in [ mix / air bubbles / in a capillary tube and ] the pouring-in machine sampling operation after single crystal formation, The manufacturing method of the colloid single crystal which is referred to as providing the colloid single crystal production containers which do not make a capillary tube produce pressure fluctuation, and \*\*\*\* which is forecited inconvenience by use of this container does not produce, or a dry colloid crystal will be provided.

[0010]In addition to solution of many of said problems, the colloid single crystal production containers which can also realize uniform desiccation of a colloid crystal are provided by setting up said container with specific construction material further again, Or the method of obtaining the high dry colloid crystal of single crystal nature will be provided by uniform desiccation by this means.

[0011]

[The means for carrying out business solution] As a result of inquiring wholeheartedly in this invention person, the cause which (i) air bubbles produce, Depending on pouring in through the needle of the syringe, the tip interface of the fluid in a narrow capillary tube, That it is what does not necessarily advance uniformly, but slowness and fastness

produce according to resistance, a wettable difference, etc. of a capillary tube, and the contamination phenomenon of air bubbles produces by this, and these phenomena, That it is a phenomenon which results from the power at the time of in short pouring in uniformly and fully not acting on a fluid, and (ii) pressure fluctuation, The power applied when sampling a syringe acted on the single-crystal--ization-processed colloidal solution in a capillary tube, and it traced that it was a phenomenon produced as a result of this serving as pressure fluctuation and influencing.

[0012]And in order not to produce these phenomena. When pouring in a colloidal solution, by pressing a colloidal solution fit rather, certainly, make it power act on a fluid, and in the case of syringe sampling after single crystal formation. The result of having advanced research to the pan wholeheartedly per [ which satisfies this requirement ] means since it was required to keep power from acting, As the structure of a pouring-in mouth was shown in drawing 3 which expanded the attachment relation between drawing 2 or its pouring-in mouth, and the tip of a pouring-in means as an entry of a pouring-in means tip part, it found out that an entrance could be widely solved by attaching a taper and setting up the back narrowly from the entrance to the back.

[0013]Namely, it is not necessary to use the pouring-in machine which attached the needle at the tip by making a pouring-in mouth into the structure which attached the taper. Only by inserting in a pouring-in mouth the syringe tip end part which attaches a needle, if it is not necessary to use a tube namely, and a pouring-in machine is described based on a syringe, in order to secure adhesion, The power which can be certainly stuck to a container and is pressed fit as a result can be certainly acted on a fluid, and power does not act on the single crystal formed in the capillary tube only by depending only on the only sampled operation from a taper on the contrary in the case of sampling.

[0014]Even if it is a thing of the structure which only presented cylindrical shape like old and is going to carry this out, the relation between a pouring-in machine tip and a pouring-in mouth, Except when it is formed so that both may be dimensionally in agreement, and it enables it to maintain adhesion relations by this. Even if it uses that which cannot collateralize adhesion namely, from which a size differs, it will interfere with press fit operation, Or both will be coincided dimensionally, and will be set up, the crystal which consists of a colloidal particle which aligned as a result of requiring power impossible in the case of sampling and this power's acting on a crystal, when being formed so that adhesion can be held will be disturbed, and it will have an adverse effect.

[0015]This invention can attain an expected aim, without



performing both wearing and desorption smoothly and producing forecited inconvenience by constituting the structure of a pouring-in mouth in taper shape at least. [0016]In the prior invention which makes this invention the premise art, pouring-in operation and a series of the operations of all the which give a shearing flow use a syringe chiefly, are performed by piston operation of the syringe, and pouring-in operation through a needle is carried out by between them. Therefore, when stating in relation to old art, based on the syringe, have described the pouring-in means chiefly, but. The aim of this invention is a passage on \*\*, and the other pouring-in means can also be used from the structure of the pouring-in mouth indicated as a means for it as well as the ability to use like old the syringe in the state where the needle was extracted, to pour in with a needle being used as unnecessary through these all the operation periods.

[0017]Namely, the meaning of a point which set the structure of the pouring-in mouth as the structure which attached the taper, It is what means using the thing of the structure corresponding to this pouring-in mouth also about the pouring-in means concerned as a matter of fact, This can be used for liquid pressure acquisition stages, such as that in which the syringe of the structure which extracted the needle as shown in drawing 2, of course, has not only this but the same structure, for example, the syringe by which the tip is presenting the taper to tapered shape, and a pipette. However, probably, about the syringe equipped with a needle, being eliminated will be clear also from the explanation on \*\*.

[0018]Anyway, compare with the conventional technology which was only performing pouring-in operation by the cylindrical shape pouring-in mouth via the needle at the tip of a syringe, and this invention, There is also no contamination of air bubbles only by the operation which only inserts in a pouring-in mouth the pouring-in machine which has the structure corresponding to this by lecturing on the composition on \*\*, It is not necessary to use a troublesome tube for handling, and a colloidal solution is pressed fit in a container as it is, Can give a shearing flow to a colloidal solution, and can form a single crystal, and again the sampling of the pouring-in machine after colloid single crystal formation only by the operation point of only sampling. Without requiring power, it can \*\*\*\*\* easily and power which disturbs this to the generated colloid single crystal does not act.

[0019]As a result of also examining wholeheartedly a means to dry uniformly the colloid crystal containing the carrier fluid confined by the (iii) container, as raw material liquid of a colloid crystal, It finds out that it can solve by using the

colloidal solution which makes water carrier fluid, and on the other hand using the thing which comes to carry out manufacture composition by the plastic which has fixed moisture permeability as a container for crystal preservation.

[0020]In the method indicated by the old prior invention, desiccation, It is carried out through the pouring-in mouth open end which is only a part when it sees from the colloid crystal accommodated in the whole container, In order to become heterogeneous and to solve this from being carried out by being extremely unevenly distributed in a part, By selecting the construction material of a container based on specific character, and choosing and combining the colloidal solution by specific carrier fluid with this, as a result of taking lessons from the means which fulfills this condition the place which needs to be dried on the whole and inquiring wholeheartedly, It finds out that the whole colloid crystal can be dried uniformly.

[0021]Namely, by selecting and using a plastic with fixed moisture permeability for a container, and combining the colloidal solution which made water carrier fluid at this, The moisture of carrier fluid is made to emit through the whole wallplate of a container from the obtained crystal, and it compares with the conventional technology by the local evaporation region which was unevenly distributed in the pouring-in mouth by this, and if it puts in another way with the same drying rate mostly over the whole container surface, on the whole, it can dry uniformly.

[0022]As stated above the 1st solving means of this invention, (1) It is a colloid single crystal production container which has a plate shaped capillary which has the pouring-in mouth provided in container both ends by protruding, is connected with this pouring-in mouth and has a gap of 0.5 mm or less, From the entrance to the back, attach the back, and the structure of this pouring-in mouth is widely, set [ a taper ] up for an entrance narrowly, and the capillary tube field of the construction material of a container at least is having considered it as the colloid single crystal production containers constituting with the transparent material.

[0023]the 2nd solving means of this invention -- (2) -- the container shown in said 1st solving means is having considered it as the colloid single crystal production containers being glass.

[0024]the 3rd solving means of this invention -- (3) -- the container shown in said 1st solving means is having considered it as the colloid single crystal production containers being a product made from a plastic.

[0025]the 4th solving means of this invention -- (4) -- it is the single crystal-ized method of the colloid crystal forming a colloid single crystal by pressing a colloidal solution fit in

said solving means 1 and 2 or colloid single crystal production containers given in 3, and giving a shearing flow to this colloidal solution.

[0026]the 5th solving means of this invention -- (5) -- it is a manufacturing method of the dry colloid crystal pressing a colloidal solution fit in said solving means 1 or colloid single crystal production containers given in 2, forming a colloid single crystal by giving a shearing flow to this colloidal solution, and giving subsequently to a drying process.

[0027]the 6th solving means of this invention -- (6) -- a colloidal solution in the colloid single crystal production containers of said solving means 3 statement, [ press fit and ] It is a manufacturing method of the dry colloid crystal forming a colloid single crystal, emitting moisture slowly by the slight moisture permeability which the plastic material subsequently has, and obtaining a dry crystal by this by giving a shearing flow to this colloidal solution.

[0028]The 7th solving means of this invention is set to obtain (7) dry colloid crystal, Without being based especially on specific structure, a colloid crystal is produced in the container made from the plastic which has fixed moisture permeability, and a colloid crystal is dried by slow discharge of the moisture using the slight moisture permeability which this container has.

[0029]It has the pouring-in mouth provided here in the 1st solving means by protruding on the matters, i.e., the container both ends, of the preceding paragraph, The point used as the colloid single crystal production containers which have a plate shaped capillary which is connected with this pouring-in mouth and has a gap of 0.5 mm or less has pointed out the technical matter which is common in the prior invention made into old art, i.e., the premise art of this invention. In the narrow plate shaped capillary, this invention pours in the colloidal solution which takes a colloid crystal state, fills, and ranks second, The reason for having made setting out of the gap into the maximum 0.5 mm by giving a shearing flow to a colloidal solution, arranging the arrangement direction of a colloidal particle and forming a colloid single crystal by this, If a gap becomes larger than this, it will specify from the shearing force in a flow becoming weak and formation of a colloid single crystal becoming difficult.

[0030]The point of having attached the taper narrowly and having set up the opening of the entrance side for the back side widely a latter matter, i.e., the structure of a pouring-in mouth, The pouring-in machine which pours a colloidal solution in a container and gives a shearing flow by this, Can use pouring-in means, such as not only the syringe in the state where the needle was taken but a syringe, a pipette,

a micropipette, etc., so that it may be shown in a pouring-in means, for example, drawing 2, to have the taper shape corresponding to the shape of this pouring-in mouth, and by this. Can stick a pouring-in machine and a container, and a colloidal solution is pressed fit in a capillary tube at high speed, without involving in air bubbles, Since can give a shearing flow to a colloidal solution, a single crystal can be formed, and the contamination of the air bubbles resulting from the syringe operation with the conventional needle can be prevented and it is operated via a tube, The operation effect which was excellent in the ability to sample easily is done so, without having an adverse effect, like the sampling of the pouring-in machine after single crystal formation also disturbs a colloid single crystal.

[0031]It sets to pour in a fluid by syringe operation which attached the needle to the capillary tube of the plate-like narrow gap about the cause which air bubbles mix, Since this pouring-in mouth is wide opened towards outside even if the needle at the tip of a means slack syringe for pouring in tends to be inserted in a pouring-in mouth and it is going to pour in a fluid, It will be in the state where a pressure is not easily applied to the fluid which flows into the capillary tube from a needle, and the entry rate of a fluid will become slow for the flow resistance of a capillary tube. or [ that how depending on which a fluid and a container wall are damp is a delicate difference by the place of the cleanliness of a container wall, and the central part of a capillary tube on the other hand ], since it differs depending on the difference in the local shape whether to be an end, The shape of the oil level of the front line of the fluid which flows in the capillary tube is in the tendency which curves reflecting the difference in such how to get wet. If the entry rate of a fluid is slow, the curve of the oil level of the front line will develop and it will result in winning air bubbles finally.

[0032]the situation which inserts the needle of a syringe in the pouring-in mouth formed cylindrical, the contamination phenomenon of forecited air bubbles tends to produce since it was what is depended on a slow pouring-in mode with a needle as shown in drawing 1, and air bubbles actually tend to mix as a result in the old art which it is making into the premise of this invention was suited. That is, it cannot be denied that there was a cause of an end also in the pouring-in system of a container. However, since between a container and pouring-in machines can be stuck by the taper part 7 if according to the container of this invention which makes a pouring-in mouth the structure which attached the taper a pouring-in mouth is the container which is presenting taper shape as shown in drawing 2 and drawing 3, It becomes possible to pour in a sample solution object at high speed compulsorily by the press fit means (piston) of a

pouring-in machine, and mixing of the air bubbles in the oil level of the front line of the fluid which flows in the capillary tube can be prevented.

[0033]And if the single crystal-ized operation which follows pouring-in operation finishes, will demount a pouring-in machine tip from a pouring-in mouth, but. Since it has contacted with taper shape at this time, and a pouring-in machine is only sampled to an opposite direction with an insert direction and the adhesion relation between a pouring-in machine tip and a pouring-in mouth can be canceled easily, a pouring-in machine can be removed easily. On the other hand, in the premise art in which the taper is not attached, If it tries to have removed a syringe from a tube since it was connected via the tube as shown in drawing 1, or when it tried to have removed the whole tube, pressure fluctuation started the capillary part, and the state of the once single-crystal-ized sample might be disturbed. That is, when removing, only by slight power being added, this serves as pressure fluctuation and gets across to a capillary tube, but since the taper is given to this invention, the removal does not give pressure fluctuation to a capillary tube only by sampling.

[0034]Since the air bubbles mixed once cannot remove this as a matter of fact, when air bubbles once mix, product value will be lost and it cannot but lay on the shelf of it with an expensive container. This invention, i.e., by lecturing on the easy means which makes structure of a pouring-in mouth tapered structure, the point that generating of air bubbles can be suppressed now has a very big meaning.

[0035]The point which used material of the container as the transparent material in the solving means of (1), (2), and (3) and the point used as glass, or the point made into the plastic, That it is necessary to conduct the optical inspection of the quality of the colloid single crystal formed in the inside of a container and, and a colloid crystal, By closing accommodated in the manufactured container, the embodiment which tends to be element-ized and it is going to use as a colloid crystal element the whole container also needs to be made with the transparent material from a certain thing, and its container is the composition for it from it. And glass and a plastic are indicating the concrete material selection for it.

[0036]Next, the solving means of (4) and (5) indicates the method of single-crystal-izing a colloid crystal using said solving means 1 and 2 or colloid single crystal production containers given in 3, and the method of producing a dry colloid crystal further. That is, a colloidal solution is pressed fit in forecited colloid single crystal production containers, in a container, a shearing flow is given to this colloidal solution, a single crystal is formed by this, and a dry colloid

crystal is produced by adding a drying process further. The drying method in this case is performed by the same method as the prior invention made into premise art by this invention. That is, the pouring-in mouth provided in container both ends by protruding is opened wide, and it is carried out by evaporating carrier fluid over many hours slowly from here.

[0037]The solving means of a statement indicates the method of producing a dry colloid crystal by a characteristic drying means to (6) and (7). this -- the former -- this -- the uniform desiccation which could not be attained depending on the drying means by a characteristic drying means is attained, and the high dry colloid crystal of single crystal nature can be obtained. In this method, the place made into the requirement matter for it, The colloidal solution to be used chooses the colloidal solution which makes water carrier fluid, and. Choose what was produced by the plastic with which the crystal production containers to be used also have fixed moisture vapor transmission, and by this the drying process after forming a single crystal, It dries by emitting moisture slowly from the whole container by the slight moisture permeability which the plastic material has chiefly, and the high dry colloid crystal of single crystal nature is obtained by this.

[0038]

[An embodiment of the invention and an example] The basic constitution of the colloid single crystal production containers of this invention and the single crystal-ized method of the colloid crystal which uses this container are explained based on drawing 2. In drawing 2, 2 is a colloid single crystal production container which has a plate shaped capillary. The container is made of a transparent material in hard. For example, plastics, such as silica glass, various optical glass, polycarbonate, and an acrylic, are mentioned. The plate-like capillary tube 5 is formed by having set the inside of a container as the gap of 0.5 mm or less. The pouring-in mouth 1 is protruded and formed in the both ends of a container, and the pouring-in mouth of the one end continues from the internal plate shaped capillary 5 to the pouring-in mouth of the other end further. 3 is a pouring-in machine and 4 is a pouring-in machine tip part.

[0039]As shown in the enlarged drawing 3, widely, in the back, the pouring-in mouth 1 attaches a taper narrowly and the opening of the entrance side is set up. Only by operation which inserts the tip of a pouring-in machine with this taper, the tip part of a pouring-in machine can stick and contact a pouring-in mouth, and removal can also perform it easily. The thing of the form corresponding to the hypodermic needle attachment portion 4 of the commercial standard pouring-in machine 3, i.e., a lure syringe, is just used for this

taper shape, and it does not need to be special especially.  
 [0040]Although the function for which a pouring-in machine is asked presses a colloidal solution fit in a container, or a shearing flow is given to a colloidal solution, performing pressure operated of making 1 shaft orientations flow along plate shaped capillary space etc. is called for and a syringe is mentioned most typically, If the above-mentioned operation is possible, it is as having mentioned above that not only a syringe but a syringe, a pipette, etc. can be used.

[0041]As shown in drawing 1 in the case of the prior invention made into the premise art of this invention, inserted the syringe with a needle into the pouring-in mouth of cylindrical shape, and were injecting the colloid crystal solution into it, but. In this case, to the sample solution which flows into the capillary tube, a pressure is hard to be applied, and since it is a flow resistance of a capillary tube, the entry rate of a fluid becomes slow. Since how depending on which a fluid and a container wall are damp differs on the other hand depending on the difference in the local shape whether to be a delicate difference, and the central part or the end of a capillary tube by the place of the cleanliness of a container wall, The shape of the oil level of the front line of the fluid which flows in the capillary tube is in the tendency which curves reflecting the difference in such how to get wet. If the entry rate of a fluid is slow, the curve of the oil level of the front line will develop and it will result in winning air bubbles finally. The probability that air bubbles will actually mix by the injection method to which the taper is not given was high.

[0042]However, since a taper part sticks by the way and it can contact when the pouring-in mouth considered it as the container which has taken taper shape as mentioned above, Since it can seal certainly between a container and a pouring-in machine, it can pour in a sample solution object at high speed compulsorily by pushing of the piston of a pouring-in machine. Before the phenomenon in which the shape of the oil level of the front line of the colloidal solution which flows in the capillary tube curves appears, it can complete pouring in quickly, and mixing of air bubbles is prevented.

[0043]Here, a colloidal particle begins organic high polymer particles, such as polystyrene and polymethylmethacrylate, here, and the oxide particle of various metal, such as metal particles, such as a silica particle, gold, and silver, titanium, and iron, etc. are mentioned further. As carrier fluid, organic solvents including water, such as various alcohol and benzene, etc. are mentioned.

[0044]The colloidal solution which takes a colloid crystal state in a pouring-in machine first is extracted, it inserts until it sticks a pouring-in machine tip to a pouring-in

mouth, and a colloidal solution is pressed fit in a container. The solution pressed fit passes through the channel crooked from the pouring-in mouth which protruded, is poured into a plate shaped capillary, and is filled with a solution to the pouring-in mouth of the other end. A colloidal solution is single-crystal-ized by giving a shearing flow to the colloid crystal in a capillary tube by pushing and lengthening the piston of a pouring-in machine by the above operation, after pressing the colloidal solution 6 fit in a container. Since it has joined together with taper shape at this time and the adhesion condition of a pouring-in machine tip and a pouring-in mouth is easily cancelable although a pouring-in machine tip is removed from a container if needed if this operation finishes, it can remove without hardly giving pressure fluctuation to the inside of a capillary tube.

[0045] Since the syringe tip and the pouring-in mouth which protruded on the container were combined and sealed by the tube when it was the former (drawing 1), when removing a pouring-in machine, i.e., a syringe, Although a possibility of disturbing the crystalline structure of the sample which pressure fluctuation got across to the inside of a capillary tube, and was single-crystal-ized with much trouble at the process drawn out from a tube was high, this inconvenience was able to be canceled by the above-mentioned means.

[0046] After a colloid single crystal is formed, by closing both the pouring-in mouth, it is element-ized and let the whole container be a colloid crystal element. Or when making a dry colloid crystal profitably like, a dry crystal is obtained by opening a pouring-in mouth wide and evaporating carrier fluid slowly.

[0047] Although the above explained how to produce a colloid single crystal and a dry crystal using the colloid single crystal production containers of this invention, By using the container made in the construction material of the container by plastics, such as polycarbonate, an acrylic, etc. which have fixed moisture permeability, and using the colloidal solution which uses water as a liquid medium as a colloidal solution, How to lead the plastic which constitutes container wall material through a container, to evaporate, and to dry water, with to obtain a dry colloid crystal is explained.

[0048] As for the quantity the character which penetrates the moisture of a plastic indicates "moisture permeability" and its value to be, "moisture vapor transmission" is usually used. Moisture vapor transmission is specified here at JIS (JIS Z 0208), and the numerical value which expresses moisture vapor transmission in this invention has pointed out the numerical value based on this regulation. Moisture vapor transmission of a plastic when thickness is 1 mm (a unit) It is expressed with the value which shows the



moisture weight which penetrates the plastic of area  $2$  of  $1\text{ m}$  over  $24\text{ hours}$  in  $\text{g/m}^2$  and  $24\text{Hr}$ ,  $40\text{ **}$ ,  $90\%\text{RH}$ , i.e.,  $40\text{ **}$ , and the environment of  $90\%$  of relative humidity. If it indicates concretely, Polycarbonate was about  $4$ , the polystyrene about  $3$  polypropylene about  $0.3$ , the high density polyethylene  $0.13$ , and Appel's (the plastic of Mitsui Chemicals, Inc.; trade name)  $0.09$  order.

[0049]The polycarbonate container which this invention person actually used in the experiment [ $1\text{ mm}$  in thickness, surface area  $2$  of about  $0.001\text{ m}$  ( $10\text{-cm}^2$ ), If the trial calculation of the drying time of a colloid crystal is made based on the above-mentioned moisture vapor transmission about the colloidal solution weight of  $100\text{g} / \text{m}^2$  to  $500\text{g} / \text{m}^2$ ] per unit area (the actual amount of solutions in a container is  $0.1\text{g}$  to  $0.5\text{g}$ ), The time which desiccation takes will be  $125\text{ days}$  from the  $25\text{th}$  calculatively. When this was contrasted with the actual dry experiment, in the dry experiment, although a dry colloid crystal is obtained, several months were required from the tenth, and by the calculation top and the actual experimental result, there is no difference like  $**$  and it was mostly in agreement.

[0050]The construction material of the container for colloid crystal formation which was suitable for manufacturing a dry colloid crystal from the experiment shown above or various data, Moisture vapor transmission when thickness is  $1\text{ mm}$  is from  $0.3\text{ g/m}^2$  and  $24\text{Hr}$ ,  $40\text{ **}$ , and a  $90\%\text{RH}$  grade. It seems that it is usable even in  $40\text{ g/m}^2$  and  $24\text{Hr}$ ,  $40\text{ **}$ , and a  $90\%\text{RH}$  grade. About a minimum, by  $0.3\text{ g/m}^2$  and  $24\text{Hr}$ ,  $40\text{ **}$ , and below  $90\%\text{RH}$ , since the time which desiccation takes becomes long too much, a mentioned range takes this into consideration. On the other hand, it is the reason prescribed about the maximum that even  $40\text{g} / \text{m} ]^2$  and  $24\text{Hr}$ ,  $40\text{ **}$ , and a  $90\%\text{RH}$  grade are from a viewpoint whether the decade grade of polycarbonate is appropriate as an operation field. However, the above-mentioned regulation is a thing which provides the meaning as one rule of thumb to the last and to carry out.

[0051]As mentioned above, although the container was set up with the plastic with fixed moisture vapor transmission and being indicated per [ which obtains a dry colloid crystal ] example using this moisture permeability, After producing a colloid single crystal in the container made from a plastic, this dry process stops the pouring-in mouth  $1$ , is performed, and is performed by settling this state for a long period of time by spreading the moisture inside a container and emitting it through the plastic which is a wallplate of a container. If it sees from a viewpoint of dry operation, the releasing speed of moisture is dramatically slow, and if it is usual, the discharge phenomenon of such

moisture can be disregarded in most cases, but in this invention, the small discharge phenomenon of moisture is very important for this extent that can be disregarded. That is, rapid evaporation gives a dry strain to the film obtained, causes a crack, or leaves an inconvenient distortion on optics, and is not preferred. Like this invention, there is dramatically little quantity of the sample in a capillary tube, according to the area of the plastic which touches this being dramatically large relatively, it can receive, sufficient time can be spent by discharge of few moisture, and a dry colloid crystal can be obtained from several weeks in about several months. This is an important technical matter, also in order to avoid inconvenience which was mentioned above.

[0052]Anyway, in the dry process performed through the whole wallplate of this container, it is dared to close the pouring-in mouth 1 of a container, and it is important here to keep moisture from evaporating quickly. If a pouring-in mouth is not closed, desiccation advances quickly from near a pouring-in mouth, it becomes uneven desiccation, and generating of defects, such as a crack resulting from the heterogeneity of the desiccation to a colloid crystal and distortion, takes place. If a pouring-in mouth is closed, from the whole surface of the capillary tube which touched the colloid crystal, removal of moisture will be performed very slowly and uniformly, and the high dry colloid crystal of single crystal nature with few defects will be obtained by this.

[0053]Although that the shape of that pouring-in mouth is taper shape may naturally contain as an embodiment the container used for enforcing this method facing the dry colloid crystal manufacturing method based on the plastic container which has the fixed moisture vapor transmission described above, There is not necessarily necessity which is a thing of the structure of this specification. Namely, in production using the moisture vapor transmission of a plastic container of a dry colloid crystal, can carry out also in the container of the usual structure and it compares with the method dry through the open end of an old pouring-in mouth, The exceptional operation effect that uniform desiccation can be carried out is done so, and the meaning as an independent invention is accepted. Claim 7 and the 7th solving means corresponding to this have indicated the technical matter of this point.

[0054]As mentioned above, if the container for obtaining the colloid crystal of this invention based on plastic material is not the thing excellent in the optical transparency in addition to the moisture vapor transmission which carried out the forecited regulation also of also excelling in transparency from the place demanded as a kind of plastic for which it was suitable, it will not become. Therefore, in

selection of a plastic. Although these things must be taken into consideration synthetically, if the plastic excellent in the comprehensive target is mentioned as a result of taking lessons from the physical properties of a plastic, etc. in this invention and inquiring minutely, generally known things, such as methacrylic resin, polycarbonate resin, and polystyrene resin, can be mentioned. namely, -- without the plastic material to be used is especially based on material with difficult acquisition -- rather -- general -- acquisition -- an easy material can be used. Since it is inconvenient if container construction material disturbs polarization in order for a colloid crystal to have a dependency in an optical property to polarization and to use this polarization dependency, from this viewpoint. The one as the characteristic of resin where a photoelastic coefficient is smaller is good, and can say that methacrylic resin is most suitable among three kinds of above-mentioned resin.

[0055]As mentioned above, although the reference indication was carried out about the conditions which obtain the dry colloid crystal based on a plastic with moisture permeability, In the case of the colloid crystal offered as an element as it is after the end of single crystal-ized operation of a colloid crystal without drying, Since it needs to be necessary to maintain the state over a long period of time, namely, it is necessary to prevent desiccation and to maintain a moisture content for preservation and plastics have moisture permeability unlike glass even when they are few when using a plastic, it must be careful of that rather. In that case, it is important that moisture vapor transmission is small (moisture vapor transmission when thickness is 1 mm as a rule of thumb. 0.3 g/m<sup>2</sup> and 24Hr, 40 \*\*, and less than 90%RH) excellent in optical transparency as construction material suitable for the mothballs of a colloid crystal. Specifically, amorphous polyolefin resin is excellent. as a concrete trade name -- APO (trade name of the company) of Mitsui Chemicals, Inc. -- the same -- Appel (trade name of the company) of the company -- ZEONEX (trade name) of Nippon Zeon Co., Ltd. is mentioned further. The moisture vapor transmission of these amorphous polyolefin system resin products has reached 0.1 g/m<sup>2</sup> and 24Hr, 40 \*\*, and below 90%RH per mm in thickness. It can be said that it is very convenient also at the point which can form the container in which each of these resin has a small photoelastic coefficient, and polarimetry is possible.

[Effect of the Invention]As mentioned above, according to this invention, removal of a pouring-in machine can be performed, without pouring of a sample being able to make mixing of air bubbles there be nothing, and giving pressure fluctuation simply, after single-crystal-izing of the colloid crystal by the pressure variation operation with a pouring-in

machine. By these effects, the quality colloid single crystal which does not have disorder by the influence of air bubbles and excessive pressure fluctuation into a capillary tube is producible. According to this invention container by the plastic which has specific moisture vapor transmission especially, since the whole sample can be mostly dried slowly homogeneously with the same drying rate, a high crystalline dry colloid crystal is obtained from the state which produced the single-crystal-ized colloid crystal. And the process is very simple and is high. [ of practicality ]

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[Translation done.]

JAPANESE

[JP,2003-212700,A]

CLAIMS DETAILED DESCRIPTION TECHNICAL  
FIELD PRIOR ART EFFECT OF THE INVENTION  
TECHNICAL PROBLEM MEANS EXAMPLE  
DESCRIPTION OF DRAWINGS DRAWINGS

[Translation done.]

\* NOTICES \*

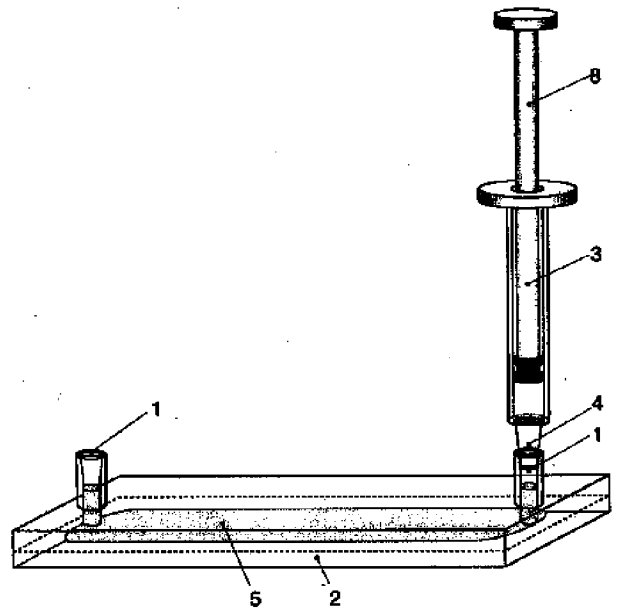
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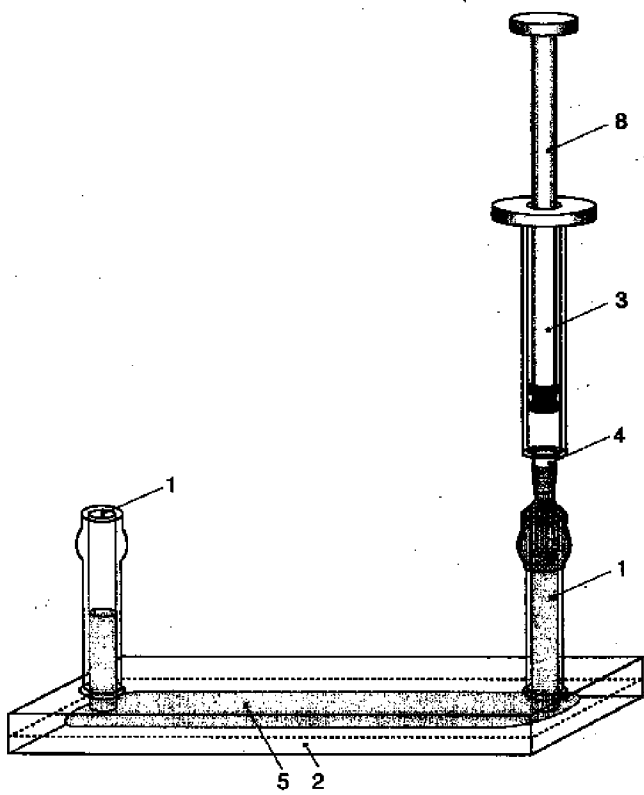
## DRAWINGS

[Drawing 1]

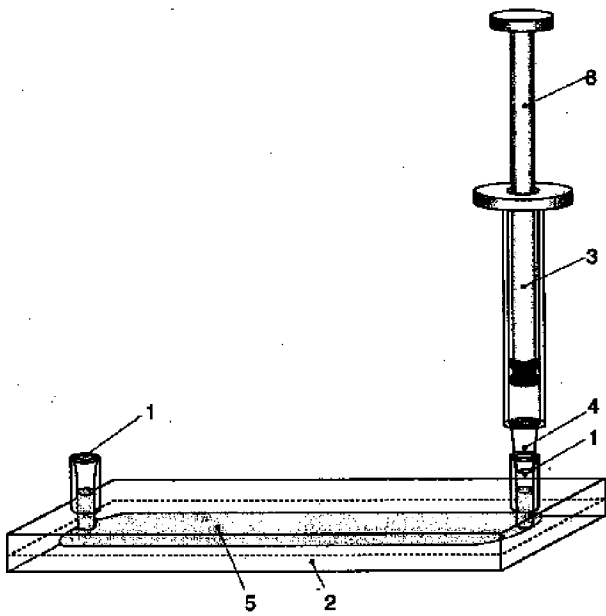
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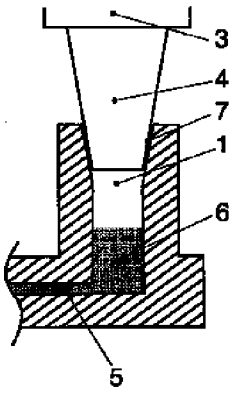
[Translation done.]



[Drawing 2]



[Drawing 3]



[Translation done.]